

PATENT COOPERATION TREATY

19.09.2000

From the
INTERNATIONAL PRELIMINARY EXAMINING AUTHORITY

To:

RUSKA & Co Oy
Runeberginkatu 5 AB
FIN-00100 HELSINKI
Finland

PCT

WRITTEN OPINION

(PCT Rule 66)

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+
PW

25.10.00

Date of mailing
(day/month/year)

26 -09- 2000

Applicant's or agent's file reference 302580/TL		REPLY DUE	within 30 days from the above date of mailing
International application No. PCT/FI99/00696	International filing date (day/month/year) 24.08.1999	Priority date (day/month/year) 24.08.1998	
International Patent Classification (IPC) or both national classification and IPC7 D 21 C 9/153			
Applicant CRS Reactor Engineering (UK) Ltd et al			

- This written opinion is the first (first, etc.) drawn by this International Preliminary Examining Authority.
- This opinion contains indications relating to the following items:

- I Basis of the report
- II Priority
- III Non-establishment of opinion with regard to novelty, inventive step and industrial applicability
- IV Lack of unity of invention
- V Reasoned statement under Rule 66.2(a)(ii) with regard to novelty, inventive step or industrial applicability, citations and explanations supporting such statement
- VI Certain documents cited
- VII Certain defects in the international application
- VIII Certain observations on the international application

- The applicant is hereby invited to reply to this opinion.

When? See the time limit indicated above. The applicant may, before the expiration of that time limit, request this Authority to grant an extension, see Rule 66.2(d).

How? By submitting a written reply, accompanied, where appropriate, by amendments, according to Rule 66.3.
For the form and the language of the amendments, see Rules 66.8 and 66.9.

Also For an additional opportunity to submit amendments, see Rule 66.4.
For the examiner's obligation to consider amendments and/or arguments, see Rule 66.4bis.
For an informal communication with the examiner, see Rule 66.6.

If no reply is filed, the international preliminary examination report will be established on the basis of this opinion.

- The final date by which the international preliminary examination report must be established according to Rule 69.2 is: 24.12.2000

Name and mailing address of the IPEA/SE Patent- och registreringsverket Box 5055 S-102 42 STOCKHOLM Facsimile No. 08-667 72 88	Telex 17978 PATOREG-S	Authorized officer Marianne Bratsberg/ELY Telephone No. 08-782 25 00
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WRITTEN OPINION

International application No.

PCT/FI99/00696

I Basis of the report

1. This opinion has been drawn on the basis of (*Substitute sheets which have been furnished to the receiving Office in response to an invitation under Article 14 are referred to in this opinion as "originally filed"*):

- the international application as originally filed.
- the description, pages _____, as originally filed,
pages _____, filed with the demand,
pages _____, filed with the letter of _____
- the claims, Nos. _____, as originally filed,
Nos. _____, as amended under Article 19,
Nos. _____, filed with the demand,
Nos. _____, filed with the letter of _____
- the drawings, sheets/fig _____, as originally filed,
sheets/fig _____, filed with the demand
sheets/fig _____, filed with the letter of _____

2. The amendments have resulted in the cancellation of:

- the description, pages _____
- the claims, Nos. _____
- the drawings, sheets/fig _____

3. This opinion has been established as if (some of) the amendments had not been made, since they have been considered to go beyond the disclosure as filed, as indicated in the supplemental Box (Rule 70.2(c)).

4. Additional observations, if necessary:

V. Reasoned statement under Rule 66.2(a)(ii) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement**1. Statement**

Novelty (N)	Claims	1-6	YES
	Claims		NO
Inventive step (IS)	Claims		YES
	Claims	1-2,5-6	NO
Industrial applicability (IA)	Claims		YES
	Claims	1-6	NO

2. Citations and explanations

Prior art cited in the International Search Report:

D1: EP 0397308 A2

D2: US 5346588 A

D3: EP 0511433 A1

D4: AT 403704 B

D5: WO 9701507 A1

The claimed invention relates to a method for the bleaching of cellulose pulp having medium consistency with ozone. According to claim 1, a stream of ozone-containing gas having an ozone concentration of at least 20% by volume is introduced into a pulp stream, without simultaneously applying high shear mixing.

The feature "of at least 20 % by volume" in claim 1 is not clear. There is inconsistency between this feature and the description. In the description of the preferred embodiments of the claimed invention on page 5 and 6, the used ozone-containing gas has an ozone concentration of 14 % by volume. This ozone concentration falls outside the subject matter claimed. See PCT International Examination Guidelines, Ch. III-4.3. Further, it is common knowledge and also stated in WO 9701507 (D5), page 4, line 35-page 5, line 2 that an ozone content as high as 20 % by volume may be spontaneously explosive. Thus, uncertainty arises whether the claimed invention is industrially applicable. Since there is no support in the description for a method for the bleaching of

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Supplemental Box

(To be used when the space in any of the preceding boxes is not sufficient)

Continuation of: V.

pulp with an ozone-containing gas with an ozone concentration of at least 20 % by volume, an amendment of the claims, so that they are in consistency with the description, is necessary.

Further, the only feature defining the invention in claim 1, besides the doubtful feature "of at least 20 % by volume", which distinguishes the claimed invention from the prior art disclosed in D1-D4, is a negative feature stating that no high-shear-mixing should be applied simultaneously as the introduction of ozone gas. The mere exclusion of the high-shear fluidising mixer is not considered to involve an inventive step in view of the cited documents and has not been shown to solve the problem with the bleaching of medium consistency pulp with ozone. Therefore, there is also doubt as to whether claim 1 specifies all the essential features needed to define the invention. In the description is stated that high-concentration, high-pressure ozone is introduced, using effective injection nozzles providing for the efficient dispersion necessary for obtaining a uniform distribution as well as sufficient mass transfer area. The need for fibre-destroying high-shear fluidising mixers, which are used in the prior art, is in this way removed. Thus, it seems necessary, in order to solve the problem with the bleaching of medium consistency pulp with ozone, without using a fluidising mixer as in prior art, to introduce an ozone-containing gas with high ozone concentration (300 g O₃/m³ or higher), at high pressure (10 bars or higher), using radially arranged injection nozzles.

Consequently, the present claims cannot be accepted.

PCT

From the INTERNATIONAL BUREAU

To:

Assistant Commissioner for Patents
 United States Patent and Trademark
 Office
 Box PCT
 Washington, D.C.20231
 ETATS-UNIS D'AMERIQUE

in its capacity as elected Office

Date of mailing (day/month/year) 21 June 2000 (21.06.00)	
International application No. PCT/FI99/00696	Applicant's or agent's file reference 302580/TL
International filing date (day/month/year) 24 August 1999 (24.08.99)	Priority date (day/month/year) 24 August 1998 (24.08.98)
Applicant DE VOS, Rolf et al	

1. The designated Office is hereby notified of its election made:

in the demand filed with the International Preliminary Examining Authority on:

22 March 2000 (22.03.00)

in a notice effecting later election filed with the International Bureau on:

2. The election was

was not

made before the expiration of 19 months from the priority date or, where Rule 32 applies, within the time limit under Rule 32.2(b).

The International Bureau of WIPO 34, chemin des Colombettes 1211 Geneva 20, Switzerland Facsimile No.: (41-22) 740.14.35	Authorized officer Manu Berrod Telephone No.: (41-22) 338.83.38
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PCT

**NOTIFICATION OF THE RECORDING
OF A CHANGE**

(PCT Rule 92bis.1 and
Administrative Instructions, Section 422)

From the INTERNATIONAL BUREAU

To:

OY JALO ANT-WUORINEN AB
Iso Rrobertinkatu 4-6 A
FIN-00120 Helsinki
FINLANDE

Date of mailing (day/month/year) 09 November 2000 (09.11.00)				
Applicant's or agent's file reference 302580/TL	IMPORTANT NOTIFICATION			
International application No. PCT/FI99/00696	International filing date (day/month/year) 24 August 1999 (24.08.99)			
1. The following indications appeared on record concerning: <input type="checkbox"/> the applicant <input type="checkbox"/> the inventor <input checked="" type="checkbox"/> the agent <input type="checkbox"/> the common representative				
Name and Address RUSKA & CO. OY Runeberginkatu 5 FIN-00100 Helsinki Finland	State of Nationality		State of Residence	
	Telephone No.		+358 9 694 9099	
	Facsimile No.		+358 9 694 9865	
	Teleprinter No.			
2. The International Bureau hereby notifies the applicant that the following change has been recorded concerning: <input checked="" type="checkbox"/> the person <input type="checkbox"/> the name <input type="checkbox"/> the address <input type="checkbox"/> the nationality <input type="checkbox"/> the residence				
Name and Address OY JALO ANT-WUORINEN AB Iso Rrobertinkatu 4-6 A FIN-00120 Helsinki Finland	State of Nationality		State of Residence	
	Telephone No.		+358 9 612 6120	
	Facsimile No.		+358 9 640 575	
	Teleprinter No.			
3. Further observations, if necessary:				
4. A copy of this notification has been sent to:				
<input type="checkbox"/> the receiving Office <input type="checkbox"/> the International Searching Authority <input checked="" type="checkbox"/> the International Preliminary Examining Authority		<input type="checkbox"/> the designated Offices concerned <input checked="" type="checkbox"/> the elected Offices concerned <input checked="" type="checkbox"/> other: Former agent		

The International Bureau of WIPO 34, chemin des Colombettes 1211 Geneva 20, Switzerland Facsimile No.: (41-22) 740.14.35	Authorized officer Beatrix Morariu Telephone No.: (41-22) 338.83.38
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PATENT COOPERATION TREATY

PCT

INTERNATIONAL PRELIMINARY EXAMINATION REPORT

REC'D 8 DEC 2000

PCT

(PCT Article 36 and Rule 70)

16

Applicant's or agent's file reference 302580/TL	FOR FURTHER ACTION	See Notification of Transmittal of International Preliminary Examination Report (Form PCT/IPEA/416)
International application No. PCT/FI99/00696	International filing date (<i>day/month/year</i>) 24.08.1999	Priority date (<i>day/month/year</i>) 24.08.1998
International Patent Classification (IPC) or national classification and IPC7 D 21 C 9/153		
Applicant CRS Reactor Engineering (UK) Ltd et al		

1. This international preliminary examination report has been prepared by this International Preliminary Examining Authority and is transmitted to the applicant according to Article 36.

2. This REPORT consists of a total of 4 sheets, including this cover sheet.

This report is also accompanied by ANNEXES, i.e., sheets of the description, claims and/or drawings which have been amended and are the basis for this report and/or sheets containing rectifications made before this Authority (see Rule 70.16 and Section 607 of the Administrative Instructions under the PCT).

These annexes consist of a total of 1 sheets.

3. This report contains indications relating to the following items:

- I Basis of the report
- II Priority
- III Non-establishment of opinion with regard to novelty, inventive step and industrial applicability
- IV Lack of unity of invention
- V Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement
- VI Certain documents cited
- VII Certain defects in the international application
- VIII Certain observations on the international application

Date of submission of the demand 22.03.2000	Date of completion of this report 30.11.2000
Name and mailing address of the IPEA/SE Patent- och registreringsverket Box 5055 S-102 42 STOCKHOLM Facsimile No. 08-667 72 88	Authorized officer Marianne Bratsberg/EÖ Telephone No. 08-782 25 00

INTERNATIONAL PRELIMINARY EXAMINATION REPORT

International application No.

PCT/FI99/00696

I. Basis of the report1. With regard to the elements of the international application:^{*} the international application as originally filed the description:
pages 1 - 6, as originally filed
pages _____, filed with the demand
pages _____, filed with the letter of _____ the claims:
pages _____, as originally filed
pages _____, as amended (together with any statement) under article 19
pages _____, filed with the demand
pages 7, filed with the letter of 25.10.2000 the drawings:
pages 1 - 1, as originally filed
pages _____, filed with the demand
pages _____, filed with the letter of _____ the sequence listing part of the description:
pages _____, as originally filed
pages _____, filed with the demand
pages _____, filed with the letter of _____2. With regard to the language, all the elements marked above were available or furnished to this Authority in the language in which the international application was filed, unless otherwise indicated under this item.
These elements were available or furnished to this Authority in the following language _____ which is: the language of a translation furnished for the purposes of international search (under Rule 23.1(b)). the language of publication of the international application (under Rule 48.3(b)). the language of the translation furnished for the purposes of international preliminary examination (under Rules 55.2 and/or 55.3).

3. With regard to any nucleotide and/or amino acid sequence disclosed in the international application, the international preliminary examination was carried out on the basis of the sequence listing:

 contained in the international application in written form. filed together with the international application in computer readable form. furnished subsequently to this Authority in written form. furnished subsequently to this Authority in computer readable form. The statement that the subsequently furnished written sequence listing does not go beyond the disclosure in the international application as filed has been furnished. The statement that the information recorded in computer readable form is identical to the written sequence listing has been furnished.4. The amendments have resulted in the cancellation of: the description, pages _____ the claims, Nos. _____ the drawings, sheet/fig _____5. This report has been established as if (some of) the amendments had not been made, since they have been considered to go beyond the disclosure as filed, as indicated in the Supplemental Box (Rule 70.2 (c)).^{**}^{*} Replacement sheets which have been furnished to the receiving Office in response to an invitation under Article 14 are referred to in this report as "originally filed" and are annexed to this report since they do not contain amendments (Rules 70.16 and 70.17).^{**} Any replacement sheet containing such amendments must be referred to under item I and annexed to this report.

INTERNATIONAL PRELIMINARY EXAMINATION REPORT

International application No.

PCT/FI99/00696

V. Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

1. Statement

Novelty (N)	Claims	<u>1-6</u>	YES
	Claims	_____	NO
Inventive step (IS)	Claims	<u>1-6</u>	YES
	Claims	_____	NO
Industrial applicability (IA)	Claims	<u>1-6</u>	YES
	Claims	_____	NO

2. Citations and explanations (Rule 70.7)

Amended claims have been filed with the letter of 25 October 2000.

The claimed invention relates to a method for the bleaching of cellulose pulp having medium consistency with ozone. A stream of ozone-containing gas having an ozone concentration of at least 20% by weight is introduced into a pulp stream via radially arranged injection devices, whereby the need for fibre-destroying high shear fluidizing mixers is removed.

Prior art cited in the International Search Report:

D1: EP 0397308 A2

D2: US 5346588 A

D3: EP 0511433 A1

D4: AT 403704 B

D5: WO 9701507 A1.

Cited documents D1-D3 disclose methods for the bleaching of pulp at medium consistency (MC) with ozone. The ozone-containing gas is introduced into the pulp suspension at high pressure. The ozone concentration is high, e.g. 20-300 g/m³ in D2. A high-shear mixer is necessary for mixing the ozone with the pulp in all these documents.

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INTERNATIONAL PRELIMINARY EXAMINATION REPORT

International application No.

PCT/FI99/00696

Supplemental Box

(To be used when the space in any of the preceding boxes is not sufficient)

Continuation of: V

The invention defined in the amended claim 1 differs from the prior art in D1-D3 in that the ozone-containing gas is introduced into the pulp stream via radially arranged injection devices without using high-shear mixers.

Document D4 discloses a process for the bleaching of pulp at high concentration (HC) with ozone without using a high-shear mixer. However, it is not obvious to transfer the teachings from HC-bleaching to MC-bleaching.

Thus, it is not considered to be obvious to a person skilled in the art to modify the known processes in D1-D4 so as to arrive at the claimed invention.

Document D5 merely shows the state of the art in the production of ozone-containing gas with high ozone content.

Consequently, the claimed invention is novel, is considered to involve an inventive step and to have industrial applicability.

PATENT COOPERATION TREATY

PCT

NOTIFICATION OF ELECTION
(PCT Rule 61.2)

From the INTERNATIONAL BUREAU

To:

Assistant Commissioner for Patents
 United States Patent and Trademark
 Office
 Box PCT
 Washington, D.C.20231
 ETATS-UNIS D'AMERIQUE

in its capacity as elected Office

Date of mailing (day/month/year) 21 June 2000 (21.06.00)	
International application No. PCT/FI99/00696	Applicant's or agent's file reference 302580/TL
International filing date (day/month/year) 24 August 1999 (24.08.99)	Priority date (day/month/year) 24 August 1998 (24.08.98)
Applicant DE VOS, Rolf et al	

1. The designated Office is hereby notified of its election made:

in the demand filed with the International Preliminary Examining Authority on:

22 March 2000 (22.03.00)

in a notice effecting later election filed with the International Bureau on:

2. The election was

was not

made before the expiration of 19 months from the priority date or, where Rule 32 applies, within the time limit under Rule 32.2(b).

The International Bureau of WIPO 34, chemin des Colombettes 1211 Geneva 20, Switzerland Facsimile No.: (41-22) 740.14.35	Authorized officer Manu Berrod Telephone No.: (41-22) 338.83.38
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ATENT COOPERATION TRE/

PCT

NOTIFICATION OF THE RECORDING
OF A CHANGE(PCT Rule 92bis.1 and
Administrative Instructions, Section 422)

Date of mailing (day/month/year) 09 November 2000 (09.11.00)

From the INTERNATIONAL BUREAU

To:

OY JALO ANT-WUORINEN AB
Iso Roobertinkatu 4-6 A
FIN-00120 Helsinki
FINLANDE

Applicant's or agent's file reference 302580/TL	IMPORTANT NOTIFICATION
International application No. PCT/FI99/00696	International filing date (day/month/year) 24 August 1999 (24.08.99)

1. The following indications appeared on record concerning:

the applicant the inventor the agent the common representative

Name and Address RUSKA & CO. OY Runeberginkatu 5 FIN-00100 Helsinki Finland	State of Nationality	State of Residence
	Telephone No. +358 9 694 9099	
	Facsimile No. +358 9 694 9865	
	Teleprinter No.	

2. The International Bureau hereby notifies the applicant that the following change has been recorded concerning:

the person the name the address the nationality the residence

Name and Address OY JALO ANT-WUORINEN AB Iso Roobertinkatu 4-6 A FIN-00120 Helsinki Finland	State of Nationality	State of Residence
	Telephone No. +358 9 612 6120	
	Facsimile No. +358 9 640 575	
	Teleprinter No.	

3. Further observations, if necessary:

4. A copy of this notification has been sent to:

<input checked="" type="checkbox"/> the receiving Office	<input type="checkbox"/> the designated Offices concerned
<input type="checkbox"/> the International Searching Authority	<input checked="" type="checkbox"/> the elected Offices concerned
<input checked="" type="checkbox"/> the International Preliminary Examining Authority	<input checked="" type="checkbox"/> other: Former agent

The International Bureau of WIPO 34, chemin des Colombettes 1211 Geneva 20, Switzerland Facsimile No.: (41-22) 740.14.35	Authorized officer Beatriz Morariu Telephone No.: (41-22) 338.83.38
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PATENT COOPERATION TREATY

PCT

INTERNATIONAL SEARCH REPORT

(PCT Article 18 and Rules 43 and 44)

Applicant's or agent's file reference 302580/TL	FOR FURTHER ACTION	see Notification of Transmittal of International Search Report (Form PCT/ISA/220) as well as, where applicable, item 5 below.
International application No. PCT/FI 99/00696	International filing date (<i>day/month/year</i>) 24 August 1999	(Earliest) Priority Date (<i>day/month/year</i>) 24 August 1998
Applicant CRS Reactor Engineering (UK) Ltd et al		

This international search report has been prepared by this International Searching Authority and is transmitted to the applicant according to Article 18. A copy is being transmitted to the International Bureau.

This international search report consists of a total of 3 sheets.

It is also accompanied by a copy of each prior art document cited in this report.

1. Certain claims were found unsearchable (See Box I).
2. Unity of invention is lacking (See Box II).
3. The international application contains disclosure of a nucleotide and/or amino acid sequence listing and the international search was carried out on the basis of the sequence listing
 - filed with the international application.
 - furnished by the applicant separately from the international application,
 - but not accompanied by a statement to the effect that it did not include matter going beyond the disclosure in the international application as filed.
 - transcribed by this Authority.
4. With regard to the title, the text is approved as submitted by the applicant.

the text has been established by this Authority to read as follows:

Bleaching of medium consistency pulp with ozone, without high shear mixing.
5. With regard to the abstract,
 - the text is approved as submitted by the applicant.
 - the text has been established, according to Rule 38.2(b), by this Authority as it appears in Box III. The applicant may, within one month from the date of mailing of this international search report, submit comments to this Authority.
6. The figure of the drawings to be published with the abstract is:

Figure No. _____

 - as suggested by the applicant.
 - because the applicant failed to suggest a figure.
 - because this figure better characterizes the invention.

None of the figures.

INTERNATIONAL SEARCH REPORT

International application No.

PCT/FI 99/00696

A. CLASSIFICATION OF SUBJECT MATTER

IPC7: D21C 9/153

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC7: D21C

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

SE,DK,FI,NO classes as above

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	EP 0397308 A2 (A. AHLSTRÖM CORPORATION), 14 November 1990 (14.11.90), page 2, line 49 - page 3, line 9 --	1-6
A	US 5346588 A (HERBERT SIXTA ET AL), 13 Sept 1994 (13.09.94), column 3, line 1 - line 49 --	1-6
A	EP 0511433 A1 (KAMYR, INC.), 4 November 1992 (04.11.92), page 4, column 4, line 1 - line 26 --	1-6
A	AT 403704 B (INGERSOLL-RAND COMPANY), 25 May 1998 (25.05.98), page 3, line 6 - line 35, claim 1 --	1-6

 Further documents are listed in the continuation of Box C. See patent family annex.

* Special categories of cited documents:	"T"	later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
"A" document defining the general state of the art which is not considered to be of particular relevance	"X"	document of particular relevance: the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
"E" earlier document but published on or after the international filing date	"Y"	document of particular relevance: the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art
"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)	"&"	document member of the same patent family
"O" document referring to an oral disclosure, use, exhibition or other means		
"P" document published prior to the international filing date but later than the priority date claimed		

Date of the actual completion of the international search

9 December 1999

Date of mailing of the international search report

14-12-1999

Name and mailing address of the ISA/
Swedish Patent Office
Box 5055, S-102 42 STOCKHOLM
Facsimile No. + 46 8 666 02 86

Authorized officer

Marianne Bratsberg/ELY
Telephone No. + 46 8 782 25 00

INTERNATIONAL SEARCH REPORT

International application No.

PCT/FI 99/00696

C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	WO 9701507 A1 (ARLEMARK, JAN), 16 January 1997 (16.01.97), page 4, line 32 - page 5, line 2 -- -----	1-6

INTERNATIONAL SEARCH REPORT
Information on patent family members

02/11/99

International application No.	
PCT/FI 99/00696	

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
EP 0397308 A2	14/11/90	SE 0397308 T3 AT 111371 T CA 2012771 A,C DE 69012563 D DE 69028797 D,T EP 0479789 A,B FI 89516 B FI 892243 A FI 915268 D JP 3040888 A JP 5500241 T RU 2025547 C US 5266160 A WO 9013344 A	15/09/94 10/11/90 00/00/00 06/03/97 15/04/92 30/06/93 11/11/90 00/00/00 21/02/91 21/01/93 30/12/94 30/11/93 15/11/90
US 5346588 A	13/09/94	AT 105599 T AT 404740 B AU 636173 B AU 6455290 A BG 51052 A CA 2028788 A,C CZ 283692 B DE 59005677 D DK 426652 T EP 0426652 A,B SE 0426652 T3 ES 2023623 T FI 102194 B FI 905327 D GR 91300078 T HR 930459 A,B JP 3152286 A LT 754 A LT 3393 B LV 10513 A,B NO 176975 B PL 164587 B PT 95718 A,B RO 107715 A RU 2044809 C SI 9012041 A TR 24891 A	15/05/94 25/02/99 22/04/93 02/05/91 15/01/93 01/05/91 17/06/98 00/00/00 13/06/94 08/05/91 31/01/95 25/08/95 20/02/95 20/03/95 31/08/94 13/09/91 30/12/93 27/09/95 31/08/97 01/07/92

INTERNATIONAL SEARCH REPORT
Information on patent family members

02/11/99

International application No.

PCT/FI 99/00696

Patent document cited in search report	Publication date	Patent family member(s)		Publication date
EP 0511433 A1	04/11/92	SE 0511433 T3		
		AT 120816 T		15/04/95
		AU 1004892 A		05/11/92
		CA 2057644 A,C		31/10/92
		DE 69108712 D,T		17/08/95
		FI 921716 A		31/10/92
		JP 5209387 A		20/08/93
		US 5411633 A		02/05/95
		US 5411634 A		02/05/95
		ZA 9200375 A		15/04/93
<hr/>		<hr/>		
AT 403704 B	25/05/98	AT 178894 A		15/09/97
		CA 2132165 A		22/03/95
		SE 9403155 A		22/03/95
<hr/>		<hr/>		
WO 9701507 A1	16/01/97	AU 708855 B		12/08/99
		AU 6323896 A		30/01/97
		CA 2224987 A		16/01/97
		EP 0835222 A,B		15/04/98
		JP 11508532 T		27/07/99
		PL 324110 A		11/05/98
		SE 9502339 D		00/00/00
		US 5950065 A		07/09/99
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PATENT COOPERATION TREATY

PCT

INTERNATIONAL PRELIMINARY EXAMINATION REPORT

(PCT Article 36 and Rule 70)

Applicant's or agent's file reference 302580/TL	FOR FURTHER ACTION See Notification of Transmittal of International Preliminary Examination Report (Form PCT/IPEA/416)	
International application No. PCT/FI99/00696	International filing date (day/month/year) 24.08.1999	Priority date (day/month/year) 24.08.1998
International Patent Classification (IPC) or national classification and IPC: D 21 C 9/153		
Applicant CRS Reactor Engineering (UK) Ltd et al		

1. This international preliminary examination report has been prepared by this International Preliminary Examining Authority and is transmitted to the applicant according to Article 36.
2. This REPORT consists of a total of 4 sheets, including this cover sheet.

This report is also accompanied by ANNEXES, i.e., sheets of the description, claims and/or drawings which have been amended and are the basis for this report and/or sheets containing rectifications made before this Authority (see Rule 70.16 and Section 607 of the Administrative Instructions under the PCT).

These annexes consist of a total of 1 sheets.
3. This report contains indications relating to the following items:
 - I Basis of the report
 - II Priority
 - III Non-establishment of opinion with regard to novelty, inventive step and industrial applicability
 - IV Lack of unity of invention
 - V Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement
 - VI Certain documents cited
 - VII Certain defects in the international application
 - VIII Certain observations on the international application

Date of submission of the demand 22.03.2000	Date of completion of this report 30.11.2000
Name and mailing address of the IPEA/SE Patent- och registreringsverket Box 5055 S-102 42 STOCKHOLM Facsimile No. 08-667 72 88	Authorized officer Telex 17978 PATOREG-S Marianne Bratsberg/EO Telephone No. 08-782 25 00

I. Basis of the report

1. With regard to the elements of the international application:^{*} the international application as originally filed the description:

pages 1-6

, as originally filed

pages _____

, filed with the demand

pages _____

, filed with the letter of _____

 the claims:

pages _____

, as originally filed

pages _____

, as amended (together with any statement) under article 19

pages _____

, filed with the demand

pages 7

, filed with the letter of 25.10.2000

 the drawings:

pages 1-1

, as originally filed

pages _____

, filed with the demand

pages _____

, filed with the letter of _____

 the sequence listing part of the description:

pages _____

, as originally filed

pages _____

, filed with the demand

pages _____

, filed with the letter of _____

2. With regard to the language, all the elements marked above were available or furnished to this Authority in the language in which the international application was filed, unless otherwise indicated under this item.
These elements were available or furnished to this Authority in the following language _____ which is: the language of a translation furnished for the purposes of international search (under Rule 23.1(b)). the language of publication of the international application (under Rule 48.3(b)). the language of the translation furnished for the purposes of international preliminary examination (under Rules 55.2 and/or 55.3).

3. With regard to any nucleotide and/or amino acid sequence disclosed in the international application, the international preliminary examination was carried out on the basis of the sequence listing:

 contained in the international application in written form. filed together with the international application in computer readable form. furnished subsequently to this Authority in written form. furnished subsequently to this Authority in computer readable form. The statement that the subsequently furnished written sequence listing does not go beyond the disclosure in the international application as filed has been furnished. The statement that the information recorded in computer readable form is identical to the written sequence listing has been furnished.4. The amendments have resulted in the cancellation of: the description, pages _____ the claims, Nos. _____ the drawings, sheet/fig _____5. This report has been established as if (some of) the amendments had not been made, since they have been considered to go beyond the disclosure as filed, as indicated in the Supplemental Box (Rule 70.2 (c)).^{**}^{*} Replacement sheets which have been furnished to the receiving Office in response to an invitation under Article 14 are referred to in this report as "originally filed" and are annexed to this report since they do not contain amendments (Rules 70.16 and 70.17).^{**} Any replacement sheet containing such amendments must be referred to under Item 1 and annexed to this report.

INTERNATIONAL PRELIMINARY EXAMINATION REPORT

International application N .

PCT/FI99/00696

V. Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

1. Statement

Novelty (N)	Claims 1-6	YES
	Claims _____	NO
Inventive step (IS)	Claims 1-6	YES
	Claims _____	NO
Industrial applicability (IA)	Claims 1-6	YES
	Claims _____	NO

2. Citations and explanations (Rule 70.7)

Amended claims have been filed with the letter of 25 October 2000.

The claimed invention relates to a method for the bleaching of cellulose pulp having medium consistency with ozone. A stream of ozone-containing gas having an ozone concentration of at least 20% by weight is introduced into a pulp stream via radially arranged injection devices, whereby the need for fibre-destroying high shear fluidizing mixers is removed.

Prior art cited in the International Search Report:

D1: EP 0397308 A2

D2: US 5346588 A

D3: EP 0511433 A1

D4: AT 403704 B

D5: WO 9701507 A1.

Cited documents D1-D3 disclose methods for the bleaching of pulp at medium consistency (MC) with ozone. The ozone-containing gas is introduced into the pulp suspension at high pressure. The ozone concentration is high, e.g. 20-300 g/m³ in D2. A high-shear mixer is necessary for mixing the ozone with the pulp in all these documents.

.../...

Supplemental Box

(To be used when the space in any of the preceding boxes is not sufficient)

Continuation of: V

The invention defined in the amended claim 1 differs from the prior art in D1-D3 in that the ozone-containing gas is introduced into the pulp stream via radially arranged injection devices without using high-shear mixers.

Document D4 discloses a process for the bleaching of pulp at high concentration (HC) with ozone without using a high-shear mixer. However, it is not obvious to transfer the teachings from HC-bleaching to MC-bleaching.

Thus, it is not considered to be obvious to a person skilled in the art to modify the known processes in D1-D4 so as to arrive at the claimed invention.

Document D5 merely shows the state of the art in the production of ozone-containing gas with high ozone content.

Consequently, the claimed invention is novel, is considered to involve an inventive step and to have industrial applicability.

PCT

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International Bureau

INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

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(54) Title: BLEACHING OF MEDIUM CONSISTENCY PULP WITH OZONE WITHOUT HIGH SHEAR MIXING

(57) Abstract

A method for bleaching medium consistency pulp with an ozone-containing gas is disclosed. The highly concentrated ozone-carrying stream is introduced into the pulp stream without mechanical mixing, or accompanied by mixing of moderate intensity. Thus, advance is taken by the rapid reaction of ozone in plug-flow conditions without the use of fiber-destroying shear forces.

BLEACHING OF MEDIUM CONSISTENCY PULP WITH OZONE WITHOUT HIGH SHEAR MIXING

Field of the invention

The invention relates to a method for bleaching medium consistency pulp with an ozone-containing gas. In particular, the invention relates to the proper utilization of the very fast reaction of ozone, by providing efficient but pulp-preserving mixing immediately on introducing a substantial amount of ozone into the pulp.

Background of the invention

A number of methods for the bleaching of pulp with ozone is known in the art. These methods have developed towards carrying out the bleaching stage with medium consistency pulp, i.e. having a consistency of about 7 - 16 per cent.

Generally, ozone bleaching of medium consistency (MC) pulp according to current practice can be described as ozone generation followed by compression before introducing the ozone containing gas into the MC pulp flow. The gas-liquid-fiber suspension is vigorously treated in one or several high shear mixers before the suspension is lead to a bleach tower. The ozone may be introduced at several points along the pulp stream. Vent gases must be treated because of excess ozone carried over.

The principle described may be a result of the application of oxygen bleaching methods. Oxygen, however, operates at a much slower rate, and the temperatures used are significantly higher than those employed in ozone bleaching.

Typical and frequent problems arise from the difficulty to keep the suspension uniform. Segregation into two-phase flow easily occurs, and the ozonisation rate drops significantly (to 1 or even 0.1 % of its optimum rate) This is a dominant problem, which may be reduced by using a higher quality ozone, resulting in less gas void and consequently less need for vigorous mixing. A typical solution in the present state of the art is the use of more than one mixer. This does not, however, eliminate the problem, and by applying more shear forces to the pulp, the strength properties of the resulting product are severely affected.

A basic problem with such mixers is the short residence time, and if mixing time is increased, undesired backmixing may occur.

After leaving the mixers, the gas-pulp suspension soon segregates into two-phase flow having a relatively small gas-liquid interface per unit volume. The chemical consequences of this are low capacity and a non-uniform bleaching result. Obvious evidence of this phenomenon is the significant ozone surplus often remaining after the bleaching stage, representing both a hazard and an economical loss.

Description of the prior art

A pulp bleaching method comprising introduction of high pressure ozone in a carrier gas into a pulp stream with vigorous mixing and subsequent removal of carrier gas is disclosed in, e.g. EP-A 511 433. The major issue of this document is the removal of gas from the pulp after injection into the mixer; the reaction is said to take place essentially within ten seconds in a vertical reaction vessel situated immediately following the fluidizing mixer. Gas at about 10-13 bar containing about 3-10 % ozone by weight (6.8 vol %) is used. Preferably, the gas-pulp mixture is carried in a horizontal path following the vertical reaction step to effect separation of the large amount of carrier gas involved.

Austrian patent application 2203/92 describes a method wherein medium consistency pulp is treated with an ozone-containing gas comprising more than 120 g O₃ / normal m³ gas (5.6 vol %) whereby the gas is introduced as fine bubbles with a low differential pressure (preferably less than 1 bar). It is considered that using gas with a high ozone content, a sufficient amount of ozone is can be suspended into the gas to achieve the desired bleaching. Further, AT 2203/92 discloses the use of mixers with or without fluidisation effects, and of an ozone reaction stage subsequent to the mixing stage, as well as additional ozone addition stages with degassing stages in between. Characteristically, the highly concentrated ozone is introduced in static mixers at several points, possibly removing the inert carrier gas (normally oxygen) between stages, and the final reaction between ozone and fiber takes place in a bleach reactor, typically of the traditional upflow tower type.

30 A common feature of several other publications disclosing ozone bleach processes for medium consistency pulp is the use of fluidizing mixers in connection with the injection

of ozone-carrying gas, and the use of subsequent, relatively extended reaction stages and gas separation.

In chemical process terms, MC ozonisation can be described as ozone molecules in a gas phase that must be transported to the vicinity of the fiber and react with the fiber or other substrates. The ozone must diffuse through the gas-liquid interface, through the liquid to the fiber. The applied mixing affects the size and the relative velocity of the gas bubbles, and also the amount of fiber-liquid interface. The rate limiting step completely dominating the interaction of ozone with the fiber material is the transport of ozone through the gas-liquid interface. The gas-liquid transfer rate in a given volume is heavily dependant on the bubble size, i.e. gas-liquid surface area $\text{m}^2 \text{ gas/m}^3 \text{ suspension}$, and on the partial pressure of ozone. Other rate limiting steps, like diffusion in the fiber material itself, are determined by the nature and the consistency of the pulp, which is dominantly affected by the temperature.

Due to its dependency on mass transfer, the reaction rate of ozone is, theoretically and empirically, first order.

Consequently, efficient process solutions must be characterized by that

- the residence time distribution (RTD) must follow a plug-flow pattern (in contrast, backmixing commonly occurs in mixers), which requires special reactor geometry to avoid backmixing e.g appropriate turbine and baffles.
- mean residence time in transfer/mixer/reactor must match transport and reaction times for complete conversion of ozone; consequently reactor diameter, shape and rotation rate of a possible turbine must match flow rate.
- all ozone should be introduced in one step.

The high gas void, i.e. the low concentration of ozone generated by most present ozone generators, limits the possibilities to improve the situation. Reduced gas void in subsequent generations of ozone generators will reduce the need for mixing and reduce energy requirements as well as the size of the equipment. Higher ozone concentrations will also increase the ozonisation rate.

Disclosure of the invention

According to the method of the present invention, high-concentration, high pressure ozone is introduced into the pulp line, whereby conditions approaching plug flow are achieved, a high concentration of ozone is reached with a mass transfer area in the suspension which is sufficient for effective delignification.

According to one aspect of the present invention, the ozone is introduced using effective injection nozzles providing for the efficient dispersion necessary for obtaining a uniform distribution as well as sufficient mass transfer area to overcome the rate-delimiting mass transfer threshold present in methods according to the prior art. Thus, the need for fiber-destroying high shear fluidizing mixers is removed.

According to another aspect of the present invention, a dynamic low to medium intensity mixer is provided in the pulp stream immediately downstream of the ozone injection site. Such a mixer delivers to the pulp stream amounts of energy which are well below fluidization energies, and does not mechanically affect the fiber.

With the aid of recent technology, as disclosed in e.g. Swedish Patent Application 9502339-6, ozone with a concentration of up to 18-20 % by volume may be generated. References to concentrations as high as 300 g O₃/Nm³ have been made in prior art publications (e.g. EP-A-426 652, priority 30.10.1989), but such concentrations have not been technically feasible until recently. Using a high ozone concentration (300 g per m³ and higher) and at high pressure (10 bars and higher) together with proper injection technique, the reaction between ozone and fiber is allowed to take place at such a rate that the subsequent use of an upflow bleach tower is not necessary. The gas pressure is obtained by using precompressed oxygen, optionally mixed with other gases or liquids (e.g. argon) to maintain a suitable conductivity for ozone generation.

Oxygen is the most common carrier gas used for ozone. Highly concentrated ozone is usually considered an explosion hazard. As the ozone generating technology has developed, the accepted limit for stable oxygen-ozone mixtures has been repeatedly pushed upwards, and it appears that no absolute concentration limit for the safe handling of ozone has yet been established. Thus, use of very high ozone concentrations may yet be possible, which further facilitates use of methods according to the present invention. According to the present invention, the concentration of ozone in the gas introduced to

the pulp stream is sufficient for achieving bleaching without any fiber-destroying mechanical impact.

The initial distribution of highly concentrated ozone into the pulp is of importance, for the selectivity, as the carbohydrate component itself may be attacked by ozone if exposed for an extended time. The absence of backmixing, as may occur in high shear mixers, and the presence of plug flow conditions counteract this phenomenon.

Description of preferred embodiments

Figure 1 shows a comparison between the changes in reaction rates against time in a prior art ozone pulp bleaching process using a medium consistency mixer, and a process according to the present invention.

Example 1

Ozone-carrying gas having a pressure of about 15 bar and an ozone concentration 14 % by volume is introduced into a medium consistency pulp line carrying 1000 tons/day via a collar of radially arranged nozzles. Preferably, the nozzles are arranged to direct the gas radially into the pulp flow, essentially in a direction perpendicular to the pulp flow. A number of nozzles sufficient for distributing the gas evenly must be used. On this production scale, 186 nozzles with an inlet diameter of maximum 1 mm may be used. A sufficient mean residence time (10-40 seconds) must be allowed before any other disturbing action to the pulp.

Example 2

A medium intensity (low-shear) mixer is adapted into the pulp stream of the previous example, preferably immediately following the gas injection site. The mixer turbine is preferably a double or multiple screw with blade angles and rotation rate balanced to maintain the plug flow residence time distribution (RTD) and giving good radial mixing efficiency. The center blade has a steeper angle than the outer screw blade. Alternatively, porous metal injector devices for introduction of ozone can be arranged peripherally or on the turbine.

Figure 1 shows a comparison between a system employing a traditional medium consistency mixer with a very high capacity for a short interval dropping rapidly to zero, compared to a system according to the invention with a moderately high capacity kept constant for a long period. The dotted line represents state-of-the-art traditional medium consistency mixer technology. The first, steep section shows the effect of the mixer with high reaction and uniform distribution. The low rate section shows the effect of the corruption of the gas-suspension interface. The reaction takes place with a nonuniform distribution and the pulp is mechanically stressed by high shear mixing.

The solid line represents a system according to the invention. Throughout the process, a moderately fast reaction is taking place in a mildly stressed pulp and with a uniform distribution of ozone.

Table 1 shows a comparison in numbers between a typical conventional MC bleaching system, a state-of-the-art system and a system according to the present invention.

15

Table 1

		Conventional	Modern	Present invention
Calculus Base	Units			
Pulp production	ton OD/day	1000	1000	1000
Consistency	%	10	10	10
Ozone pressure	bar	9	9	15
Ozone concentration	w%	10	14	20
	vol%	7	10	14
Ozone charge (3-5)	kg/ton OD pulp	5	5	5
Ozone generator	kg/h	208	208	208
Ozone volume flow	m ³ /s			0,0146
Nozzle diameter	m			0,001
Number of nozzles				186
Process				
Process temperature	°C	40	40	40
Process pressure	bar	7	7	15
Pulp Flow	ton OD pulp /h	42	42	42
Volume Flow	m ³ /h MC pulp	375	375	375
Ozone gas charge	m ³ /h at actual press.	234	165	53
Gas void *	%	38	31	12
Equipment		Ozone compressor 1-3 mixers Bleach tower	Ozone compressor 1+ mixers Bleach tower	No ozone compressor No mixer Small bleach reactor

* Note: Gas void is proportional to process problems

Claims:

1. Method for bleaching of cellulose pulp having medium consistency, comprising the introduction into the pulp stream of a stream of ozone-containing gas generated from pressurized oxygen or a mixture of pressurized oxygen with at least one gas or liquid and having an ozone concentration of at least about 20 % by volume, without simultaneously applying high shear mixing.
2. A method according to claim 1, wherein the introduction of ozone is carried out at a pressure of at least 10 bar.
3. A method according to claim 1 or 2, wherein said ozone-containing gas is introduced via at least two nozzles adapted to direct the gas into the pulp stream.
4. A method according to claim 3, wherein said nozzles are adapted to direct the gas in a direction essentially perpendicular to the pulp stream.
5. A method according to any claim 1-4, wherein following gas injection the pulp stream is fed to a dynamic low to medium intensity mixer.
6. A method according to any claim 1-5, wherein ozone-carrying gas is introduced by means of porous metal injector members.

1/1

Conversion of ozone

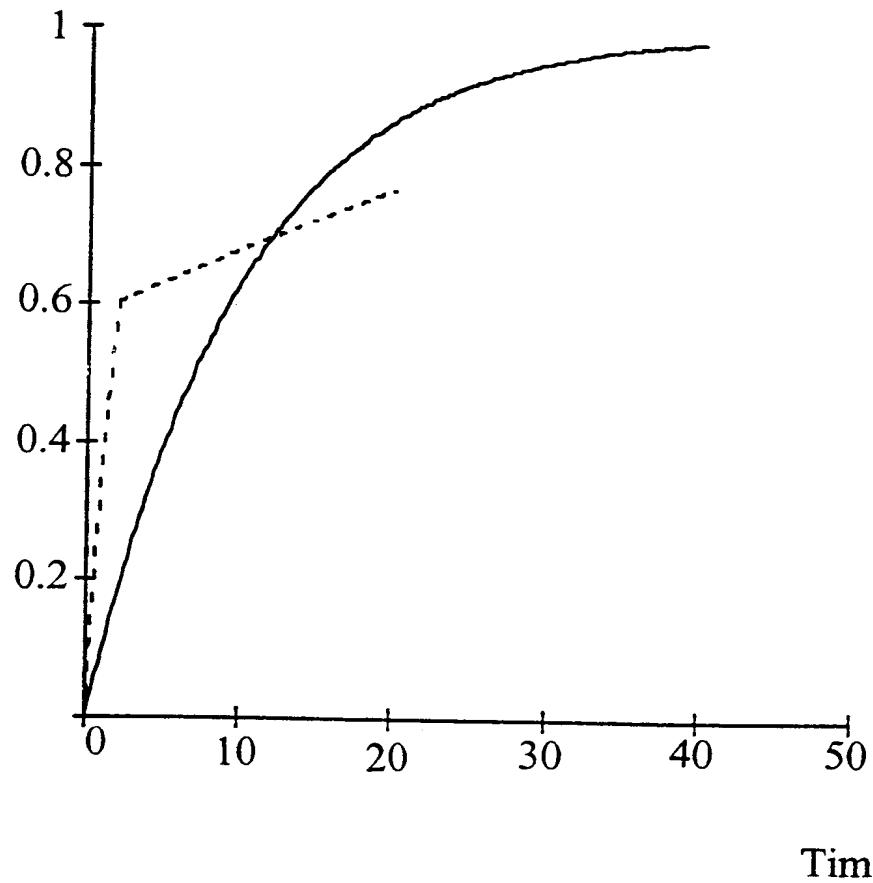


FIG. 1

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Codes used to identify States party to the PCT on the front pages of pamphlets publishing international applications under the PCT.

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1. Method for bleaching of cellulose pulp having medium consistency without using high-shear mixers, comprising the introduction into the pulp stream via radially arranged injection devices of a stream of ozone-containing gas generated from pressurized oxygen or a mixture of pressurized oxygen with at least one gas or liquid and having an ozone concentration of at least about 20 % by weight.
5
2. A method according to claim 1, wherein the introduction of ozone is carried out at a pressure of at least 10 bar.
- 10 3. A method according to claim 1 or 2, wherein said ozone-containing gas is introduced via at least two nozzles adapted to direct the gas into the pulp stream.
4. A method according to claim 3, wherein said nozzles are adapted to direct the gas in a direction essentially perpendicular to the pulp stream.
15
5. A method according to any claim 1-4, wherein following gas injection the pulp stream is fed to a dynamic low to medium intensity mixer.
- 20 6. A method according to any claim 1-5, wherein ozone-carrying gas is introduced by means of porous metal injector members.

1. Method for bleaching of cellulose pulp having medium consistency without using high-shear mixers, comprising the introduction into the pulp stream via radially arranged injection devices of a stream of ozone-containing gas generated from pressurized oxygen or a mixture of pressurized oxygen with at least one gas or liquid and having an ozone concentration of at least about 20 % by weight.
- 5 2. A method according to claim 1, wherein the introduction of ozone is carried out at a pressure of at least 10 bar.
- 10 3. A method according to claim 1 or 2, wherein said ozone-containing gas is introduced via at least two nozzles adapted to direct the gas into the pulp stream.
- 15 4. A method according to claim 3, wherein said nozzles are adapted to direct the gas in a direction essentially perpendicular to the pulp stream.
5. A method according to any claim 1-4, wherein following gas injection the pulp stream is fed to a dynamic low to medium intensity mixer.
- 20 6. A method according to any claim 1-5, wherein ozone-carrying gas is introduced by means of porous metal injector members.

AMENDED SHEET

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Ihr ZeichenOur ref. 34205/TL/th
Unser Zeichen SENT BY TELEFAX ONHelsinki,
25 October 2000Patent- och registreringsverket
Box 5055 / Valhallavägen 136
S-102 42 STOCKHOLM
SVERIGE**Patent Application No. PCT/FI99/00696 - CRS Reactor Engineering (UK) Ltd**

In response to the Written Opinion dated 26.9.2000, we submit new claim 1 to replace original claim 1.

In the table on page 6 of the description, the process according to the invention is characterised by an ozone concentration of 14 per cent by volume, corresponding to 20 per cent by weight. This also corresponds to slightly less than 300 g/Nm³ (about 270). In new claim 1, the ozone concentration is specified as 20 % by weight. It is further specified, that the ozone-containing gas is introduced by means of radially arranged injection devices, in order to include e.g. the porous metal members mentioned on page 7 and in claim 6.

As pointed out on page 4, line 25 onward of the description, the limit for safe handling of ozone has been pushed upward as technology has developed. Ozone up to 100 % is handled today using specialized cryodistillation technology. Ozone is, admittedly, highly reactive and must be stored under controlled conditions to avoid decomposition. However, in a process according to the invention, ozone need not be stored but is utilised as it is produced. Further, the pressure increase due to ozone decomposition (into biatomic oxygen) is in the worst case 10-15 % during one second. This can be handled using high pressure steel equipment, the mass of which is also high in relation to the gas volume within, so that reaction heat is readily absorbed. Conventional ozone generators comprising glass parts are naturally not feasible.

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Oy JALO ANT-WUORINEN Ab

Thus we conclude, that with state-of-the art ozone generating technology (which as such is outside the scope of the present application), the process according to the invention is industrially applicable.

We would appreciate a further opportunity to present our arguments, should the examiner have further objections against the present claims.

Yours faithfully

Oy Jalo Ant-Wuorinen Ab



Tord Langenskiöld